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Shape Forming via Colloidal Isopressing: Reformulating a Commercial Silicon Nitride Slurry with a Commercial Silane

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Abstract

Colloidal Isopressing is a new shape forming method that requires a pre-consolidated slurry with a flow stress much less than that of a commercial throwing clay (< 0.1 MPa), so that it can be injected into a rubber cavity and isopressed. It is shown that the desired interparticle pair potential can be achieved with a commercial silane for a commercial Si_3N_4 aqueous slurry (NT 154-X12) containing densification aids. Unlike previous work, this silane has a relatively short molecular length and unlike previous results, the addition of salt was not needed to shorten the molecule and achieve the rheological behavior after pre-consolidation. The results of this study also show that the transition from a fluid-like to elastic-like behavior after consolidation was related to the relative density achieved during the pre-consolidation step, which was dependent on the consolidation pressure.

1 Introduction

Colloidal Isopressing is a new shape forming method [1-4] that starts with a dispersed slurry (particles are highly repulsive) at a modest volume fraction of powder so that it can be quickly passed through a filter to remove strength degrading heterogeneities greater than a given size (e.g., $5 \mu\text{m}$). Once heterogeneities are removed, the slurry is reformulated in a second step so that the particles form a weakly attractive network. In a third step, the volume fraction of the slurry is increased by pressure filtration, a step that is known as pre-consolidation. When the

weakly attractive network is properly formulated and the consolidation pressure does not exceed a critical value, the consolidated body can be fluidized. The rheology of the pre-consolidated slurry is very important. Not only must the relative density of the pre-consolidated slurry be as high as possible for a given powder, but it must also be fluid-like so it can be injected into a rubber cavity. In the fourth step, the fluid-like, pre-consolidated slurry is injected into a rubber cavity that contains a liquid absorbing material, such as dry powder. During isopressing the relative density of the powder is increased further and the pre-consolidated, fluid-like body is converted into an elastic body that can be removed from the rubber cavity without shaped distortion.

During isopressing the relative density is further increased as liquid is transferred from the pre-consolidated slurry to the adsorbent material. The period required depends on the path length for fluid flow and other factors that are related to Darcy's Law [3]. In previous studies with a 0.2 μm alumina powder, [1,3] approximately one minute is needed to consolidate an alumina bar with a path length (bar length) of approximately 5 cm at a pressure of 100 MPa. In this same study, the volume fraction of the pre-consolidated slurry was 0.57 and increased to 0.63 during isopressing. Isopressing at higher pressures (300 MPa) produced the same relative density. Although the increase in relative density is small compared to isopressing dry powders, the rubber cavity regains its initial volume when the pressure is released, and thus, the isopressed body separates from the rubber cavity.

Studies [5] have also shown that since the consolidation pressure is larger than the capillary pressure present during drying and/or no further increase in relative density occurs for an isopressure between 100 MPa and 300 MPa, the isopressed body, still saturated with liquid, does not shrink during evaporative drying. Since shrinkage is nil, no stresses will arise during evaporative drying. These same studies showed that the isopressed body could be directly heated to the densification temperature provided that the heating rate did not exceed the rate where the water vapor became explosive. This finding has a great impact on the economics

More recently it was demonstrated [4] that when the surface of the rubber cavity was patterned, the ceramic filled all the channels to produce a patterned, ceramic surface, even when the patterning was carried out with a photolithography technique to produce channel depths at the micron scale.

To implement Colloidal Isopressing, one needs to formulate a slurry with a special interparticle pair potential that allows pre-consolidation to a high relative density, yet also produces a pre-consolidated slurry with a fluid-like rheology. Previous studies have shown that one can develop the interparticle potential either via the electrostatic double layer approach (e.g., through pH control and adding salt) [6], or as discussed in this paper, through the steric approach (i.e., by attaching linear molecules to the surface of each particle). As indicated above, the slurry is first formulated so that the particles are repulsive to allow heterogeneities to be removed by filtration. For the electric double layer approach, dispersed slurries are formulated at a pH far from the iso-electric point (the pH where the particle surface is neutral). After filtration, salt is added to produce a weakly attractive network that can be pre-consolidated to a high relative density and to produce a pre-consolidated body that can be easily fluidized.

For the steric approach, the slurry is formulated with a sufficient addition of molecules to cover the surface of the particles. [6] One end of the molecules reacts and bonds with the –metal-OH surface sites to form a ‘molecular’ brush on the particle surface. The other end is water loving, rendering the molecule hydrophilic, and allows the formulation of an aqueous slurry. A repulsive potential is produced between two particles when the molecules on one surface begin to touch those on the surface of an approaching, second particle. Repulsion arises because the molecules on one surface confine the movement of molecules on the adjacent surface, and vice versa. The confinement decreases the entropy of the system, increases the free energy, and thus produces a repulsive potential (and force) when the particles are pushed together. In effect, the molecule ‘shield’ all or a portion of the van der Waals attractive potential. When the molecules are long (e.g., > 2 nanometers), they can completely shield the van der Waals attractive potential, and thus, the particles are repulsive and produce a dispersed slurry. When the molecules are shorter, they only ‘shield’ a portion of the van der Waals potential. In this case, the particles are initially attractive and move together. But before the particles can touch one another, the short molecules on the approaching surfaces will touch first to produce the desired repulsive potential. In effect, the short-range repulsive potential produced by the short molecules and the pervasive, attractive van der Waals potential give rise to a potential well in which the particles ‘sit’ at an equilibrium separation distance.

Because the particles sit in a potential well, force must be applied to separate the particles. This force, times the number of particles per unit volume governs the yield stress of the slurry

and the yield stress of the consolidated body, provided that the particles are not pushed into contact during pressure consolidation. In addition the potential well also provides a strong repulsive force that counters the forces that try to push the particles into contact during pressure consolidation. Thus, it is possible to formulate a slurry with a short-range repulsive potential that allows pressure consolidation (e.g., via pressure filtration) without pushing too many of the particles into contact. Particles pushed into contact produce a very strong network which is difficult to pull apart without fracturing the body. Thus, during pressure consolidation, both the number density and the number of touching particles increases with the applied pressure. Franks and Lange [7], who formulated slurries with long- and short-range repulsive potentials using the electrostatic, double layer approach, showed that a critical consolidation pressure exists. Below the critical pressure, only a few particles are pushed into contact. After consolidation, the body appears elastic, but can be easily fluidized by vibration to exhibit a fluid-like rheology indicative of the short-range repulsive potential. The vibration breaks apart the strong, but minor network of touching particles. When the consolidation occurs above the critical pressure, a large majority of the particles are pushed into contact, and the body becomes elastic, namely, its yield stress is so high that it fractures before it can flow via plastic deformation.

The Colloidal Isopressing method was a technical outcome of this scientific understanding. Namely one formulates a slurry and consolidates it at a low pressure to increase the relative density while maintaining the weakly attractive particle network that exhibits fluid-like rheology. Isopressing at high pressure simply increases the relative density and pushes many particles into touching contact to produce an elastic body. An elastic body is needed to maintain the shape formed during isopressing.

In a previous study, [2] it was shown that a commercial (former Ceramics Division, Allied Signal, Inc.) aqueous Si_3N_4 slurry containing densification aids could be reformulated at pH 9 by coating the particles with water soluble molecules known as PEG-silane (N - [(triethoxysilyl)propyl]-O -polyethylene oxide urethane) shown in Fig. 1. The alkyl (RO-) groups at one end of the molecule hydrolyze in water and then react with the silanol groups (-Si-OH) on the surface of the Si_3N_4 to form a bonded molecular 'brush' on the particle surfaces. Because the PEG-silane is a relatively long, linear molecule (> 12 carbon units) it produces a net potential that is repulsive, and a dispersed slurry. Because long molecules can be made to 'look' and act shorter by adding salt to the slurry, the slurry was reformulated by adding 0.5 M of

tetramethylammonium nitrate to produce the weakly attractive particle network required for Colloidal Isopressing. Using this molecule, it was shown that both dispersed and attractive particle networks could be formed and shapes could be produced with the Colloidal Isopressing method with a relative density of 0.60. In addition, a previous study [8] had shown that the PEG-silane acts as a paint and protect the silicon nitride particle from a undesirable water reaction.

Although PEG-silane was found to be useful, its cost is prohibitive for commercial ceramic processing. For this reason, other industrial silanes were sought for this processing function. As shown below, glycidoxypyrpyltrimethoxy silane, with the commercial Dow Corning name, Z60/40, was a successful substitution. As shown in Fig. 1, its is a much shorter molecule, and as reported below, does not produce the Newtonian behavior as shown for PEG-silane. None the less, it does produce a slurry with a low enough viscosity that can be filtered to remove heterogeneities. In addition, it is shown that added salt is not required to produce the weakly attractive network required for shape forming via Colloidal Isopressing.

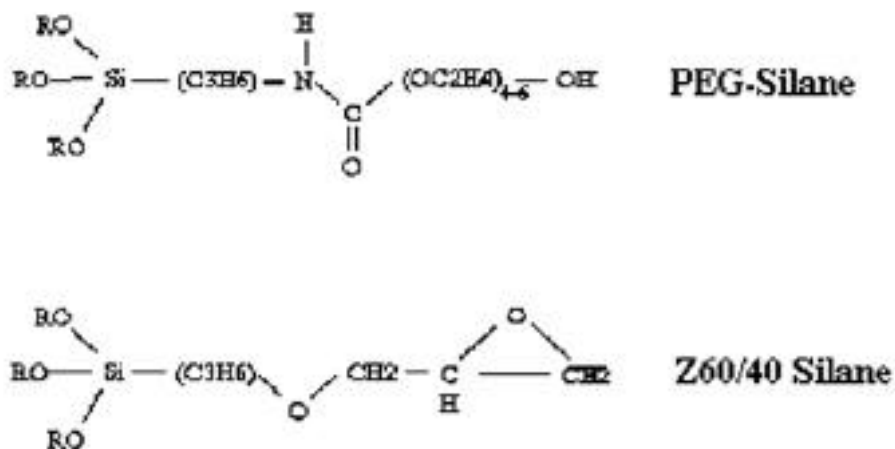


Figure 1 Configuration of the two silane molecules used for formulating silicon nitride powders for the Colloidal Isopressing shape forming method. PEG-silane was used by Reto et al. [2]; Dow Corning's product, Z60/40, a shorter molecule, was used in the current study.

2 Experimental Procedure

The aqueous slurry used for the experiments was provided from the St.-GobainCermaics and Plastics Northboro R&D Center. It contained 0.20 volume fraction of NT-154 silicon nitride powder and proprietary densification aids. The organofunctional alkoxy silane named

Glycidoxypropyltrimethoxy silane (Z60/40 from Dow Corning Co. Midland, MI) was added (2 wt % relative to powder) to the as-received slurry, sonicated with an ultrasonic horn (Model W-380, Heat Systems Ultrasonics Inc., Piscataway, NJ) and allowed to react with the powder surface for several hours on a roller mill. Before experiments were carried out with the slurry, the pH was adjusted to 10, if necessary, by adding tetramethylammonium hydroxide (TMAOH). Slurries were also formulated to contain excess salt by adding different amounts of tetramethylammonium nitrate (TMAOHNO_3 , Sigma Aldrich, Milwaukee, WI). As-received slurries (without the Z60/40 silane) were also characterized with and without added salt.

The particle size distribution was measured by dynamic light scattering (Microtrac Ultrafine particle analyzer UPA 150, Leeds and Northrup, North Wales, PA). Viscosity vs shear rate measurements were carried out with a dynamic stress rheometer (ARES, Rheometrics Inc., Piscataway, NJ) using a cup (34mm diameter) and couette (32mm diameter) cell for variable shear rates between 0.01 and 1000s^{-1} .

Slurries formulated with the Z60/40 silane with and without added salt were consolidated via pressure filtration in a pressure range of 1 to 10 MPa using identical procedures described elsewhere [2]. The relative density of the consolidated bodies were determined in the saturated state using the weight difference method. [1-4] Bodies that exhibited plastic behavior could be fluidized either by squeezing the consolidated body between the fingers, or with the use of a vibrating table. To better characterize the plastic or elastic behavior of the consolidated bodies, uniaxial compression tests between two metal plates were carried out on the consolidated bodies. The bodies were kept in plastic bags with a wet paper towel to prevent them from drying. A displacement rate of 0.3mm/min was applied and the load measured with a 100kN load cell (Instron 8562, Canton, MA) until the initial height of the bodies were reduced by $>10\%$. The nominal stress was calculated by dividing the load by the cross sectional area of the specimen. It was assumed, that the cross sectional area does not change during the test. To obtain the engineering strain, the displacement was divided by the initial height of the specimen.

The fluidized bodies were injected into a rectangular rubber cavity and prepared for isopressing (Model No. IP-2-22-60, Autoclave Engineers Inc., Erie, PA). An estimated $1/3$ of the cavity was filled with dry NT-154 powder to absorb excess water during isopressing. A filter paper between the powder and the fluidized body prevented the powder from “contaminating”

the body. Isostatic pressures from 200-300MPa were applied for 1-2min. The isopressed bars were injected from the mold and its relative density determined by the weight difference method.

3 Results

The particle size distribution of ‘as received’ NT-154 silicon nitride powder is shown in Table 1. Prior to the measurement, the slurry was diluted in water to approximately 0.1Vol.% while maintaining the pH at 10. The mean size is nearly the same as measure by St. Gobain, whereas the measurement at UCSB showed a tighter distribution.

Table 1: Particle Size Distribution for NT-154 Silicon Nitride Powder

Particle Size Distribution	D ₉₀ in μm	D ₅₀ in μm	D ₁₀ in μm
Reported by St. Gobain	1.47	0.7	0.26
Measured	1.067	0.680	0.346

Several rheology measurements were performed on the as received slurry, slurry with salt, slurry with added 2 wt % Z60/40 silane and slurry with 2 wt % Z60/40 silane + salt. All viscosity versus strain rate data were obtained for slurries formulated at pH 10. Figure 2 shows that the as-received slurry without silane and salt displayed shear-rate thinning behavior, i.e., a viscosity that decreased with increasing shear-rate. Shear rate thinning behavior is indicative of an attractive particle network. Small additions of salt increased the viscosity, i.e., increased the strength, of the attractive network. An addition of 2wt.% Z60/40 silane to the as received slurry lowered the viscosity, i.e., decreased the strength of the still attractive network; for shear rates greater than 10s^{-1} the slurry appeared to exhibit Newtonian behavior.

Figure 2 Viscosity vs shear rate for NT 154-X12 St. Gobain slurry measured ‘as received’, and reformulated with either added salt (tetramethylammonium nitrate) or the Z60/40 silane.

Figure 3 reports the viscosity behavior when different amounts of salt were added to a slurry containing 2wt.% Z60/40 silane. The addition of the salt is expected to make the silane molecules shorter, which would decrease the equilibrium separation distance, deepen the potential well, require a larger force to separate the particles, and thus make the attractive

network stronger as observed with the increase in viscosity. The ‘bump’ in the data between 0.1-1 rad/sec is indicative of slurry sliding at the interface of the couette cell; this phenomenon occurs for very thick (high viscosity) slurries.

Figure 3 Viscosity vs shear rate for NT 154-X12 St. Gobain slurry reformulated with Z60/40 silane, and containing different concentrations of added salt (tetramethylammonium nitrate).

Figure 4 reports the relative density of the saturated bodies consolidated from two different slurries at pH 10. One formulated with only the 2 wt % Z60/40 silane, and the other, also formulated with the 2 wt % Z60/40 silane, but also containing 0.1 M of the TMANO salt. Consolidation was carried out via pressure filtration at pressure between 1 MPa and 10 MPa. The consolidation of the slurry also formulated with the silane, but with a larger salt concentration was not studied in a systematic way since the strength of its particle network, via viscosity data, appeared identical to that with the smaller salt concentration. All bodies consolidated with slurries formulated with the silane and added salt had a low relative density; this was expected because the particle network was too strong to allow complete particle rearrangement during consolidation. All of these bodies, placed in a plastic bag to prevent evaporation, could be easily fluidized by pressing between the fingers, but, because of their low relative density, were not further consolidated by Colloidal Isopressing.

Bodies consolidated with slurries formulated with only the Z60/40 not only consolidated to higher relative densities, but for bodies consolidated at 7 MPa, could also be fluidized by pressing between the fingers. More qualitative rheological data were obtained for these consolidated bodies with axial compressive data shown in Fig. 5. The stress-strain data for four of these bodies illustrate that the flow stress increases with consolidation pressure. The body consolidated at 10 MPa appears to flow, but actually fractured into pieces. These data suggest that the transition from plastic to elastic behavior occurred between 7 MPa and 10 MPa. Thus, bodies consolidated at 7 MPa had a high relative density and could be fluidized for Colloidal Isopressing.

Figure 4 Per cent relative density vs consolidation pressure for two different NT 154-X12 St. Gobain slurries formulated with 2 wt % Z60/40 silane. One contains added salt (tetramethylammonium nitrate). All bodies consolidated at or below 7 MPa could be fluidized and were suitable for Colloidal Isopressing.

Fig. 5 Stress-strain behavior of 4 pre-consolidated cylinders placed in axial compression. All bodies were consolidated from the same silicon nitride slurry formulated with 2 wt % Z60/40 silane, but consolidated at different pressures (shown).

A series of pre-consolidated bodies were fluidized within a sealed, plastic bag. A corner of the bag was cut and the fluidized, pre-consolidated slurry was squeezed into a syringe, then injected into a rectangular, rubber cavity containing porous media. Isopressing was carried out between 200 MPa to 300 MPa for periods up to 2 minutes. As shown, no significant differences in relative density were observed for bodies isopressed with slurries pre-consolidated at 3 MPa and 5 MPa. The flow stress of the slurries pre-consolidated at 7 MPa were higher, and the relative density after isopressing were higher. Those isopressed at the lowest pressure, 200 MPa were highest for unknown reason(s). No shrinkage was measured during evaporative drying.

Table 2: Relative Density of Isopressed Bars Still Saturated with Water

Pre-Consolidation Pressure (MPa)	Isostatic Pressure (MPa)	Iso-Pressure Period (min.)	Good for Isopressing	Relative Density (after Isopress)
3	200	2	Yes	0.604
3	300	2	Yes	0.601
5	200	2	Yes	0.601
5	300	2	Yes	0.606
7	200	2	OK	0.634
7	300	2	OK	0.617
7	200	1	OK	0.637
7	300	1	OK	0.616

4 Discussion

The study shows that the commercial Z60/30 silane is effective molecule to produce the interparticle pair potential required to implement the Colloidal Isopressing shape forming method. Unlike the previously studied PEG-silane, which required the addition of salt to shorten the molecule in order to produce the required weakly attractive interparticle potential, the Z60/30 silane molecule was already short enough; thus, slurries formulated with Z60/30 Although this short molecule does not produce Newtonian rheology over the entire shear rate regime, it is long enough to produce a viscosity, when formulated at 20 volume %, that is low enough to allow it to be passed through a filter to remove strength degrading heterogeneities.

When salt was added to the slurry formulated with the Z60/30 silane, as expected, the particle network was too strong and did not consolidate to a high relative density. It was therefore unsuitable for further studies related to Colloidal Isopressing.

Finally, unlike particle systems where the interparticle potential is controlled by a double layer (electrostatic, double layer approach), where the plastic to elastic transition during consolidation appears to be independent on particle packing density, but only on the consolidation pressure, [7] the current work suggests that the plastic to elastic transition during isopressing is dependent relative consolidation density) as well as on the strength of the bonds (interparticle potential). A higher number of bonds per unit volume seemed to have a positive effect on the green density, as the green density of the body consolidated at 7MPa is slightly higher than those consolidated at 3MPa and 5MPa.

5 Conclusion

A commercial silicon nitride slurry (St.-Gobain Ceramics and Plastics, Northboro R&D Center, NT 154-X12) containing densification aids could be reformulated with 2 wt.% Z60/40 silane (Dow Corning, Inc.) to produce a weakly attractive particle network that is suitable for shape forming via the new Colloidal Isopressing method. The slurry, pre-consolidated at 7MPa had a relative density of 0.51, could be fluidized to fill a rubber cavity, and isopressed to form bars within 2 minutes via isopressing at 200 MPa. Isopressing increased the relative density of the saturated body to 0.62.

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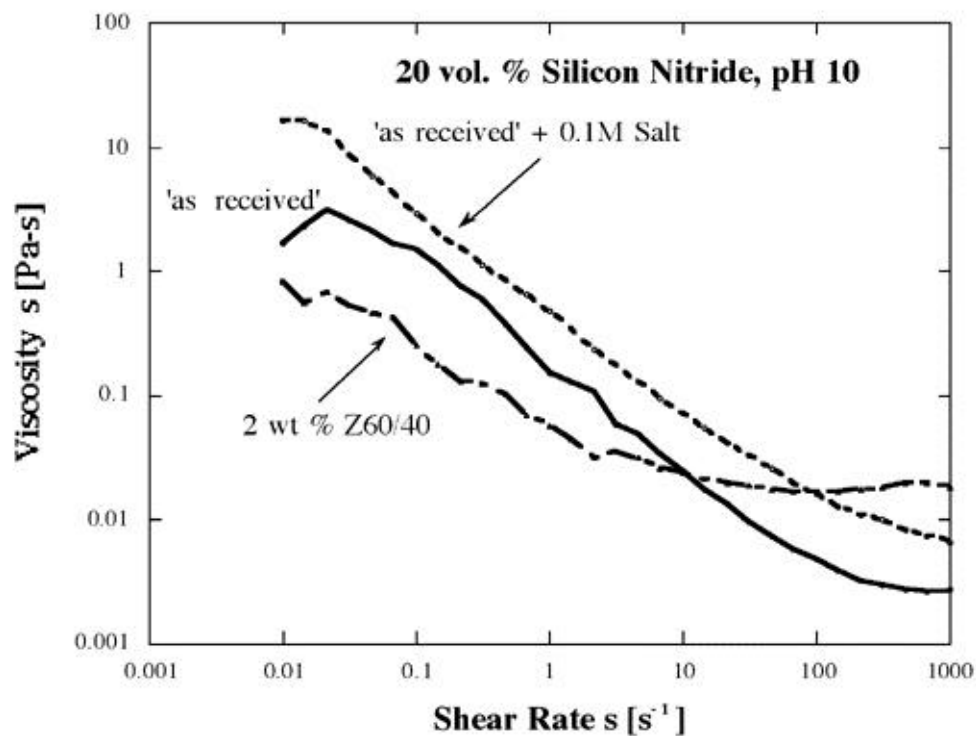


Figure 2 Viscosity vs shear rate for NT 154-X12 St. Gobain slurry measured 'as received', and reformulated with either added salt (tetramethylammonium nitrate) or the Z60/40 silane.

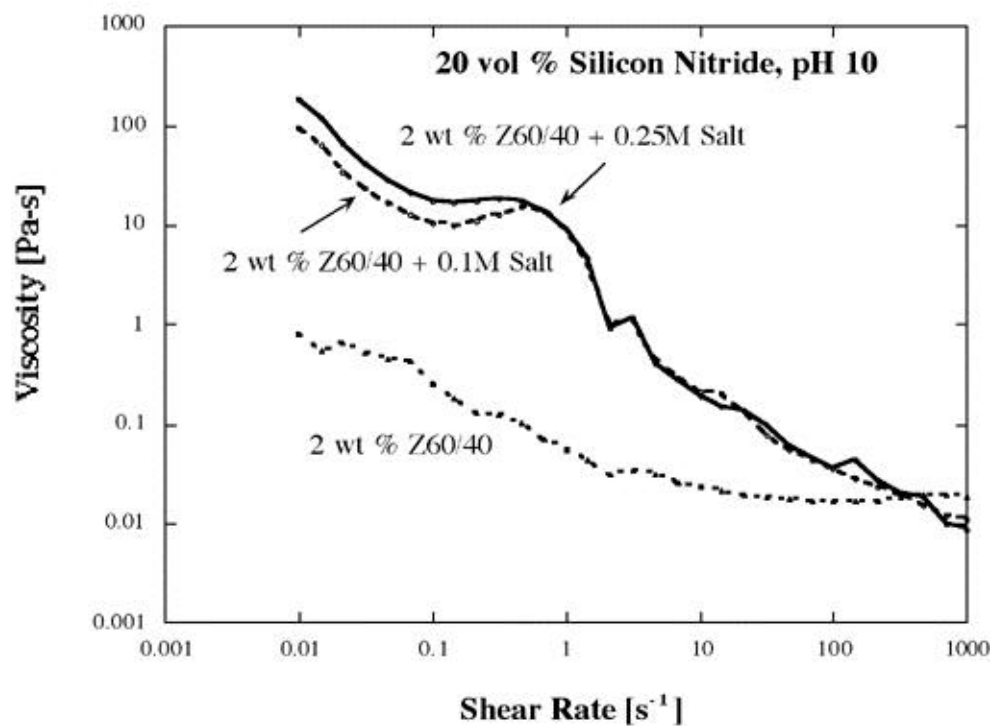


Figure 3 Viscosity vs shear rate for NT 154-X12 St. Gobain slurry reformulated with Z60/40 silane, and containing different concentrations of added salt (tetramethylammonium nitrate).

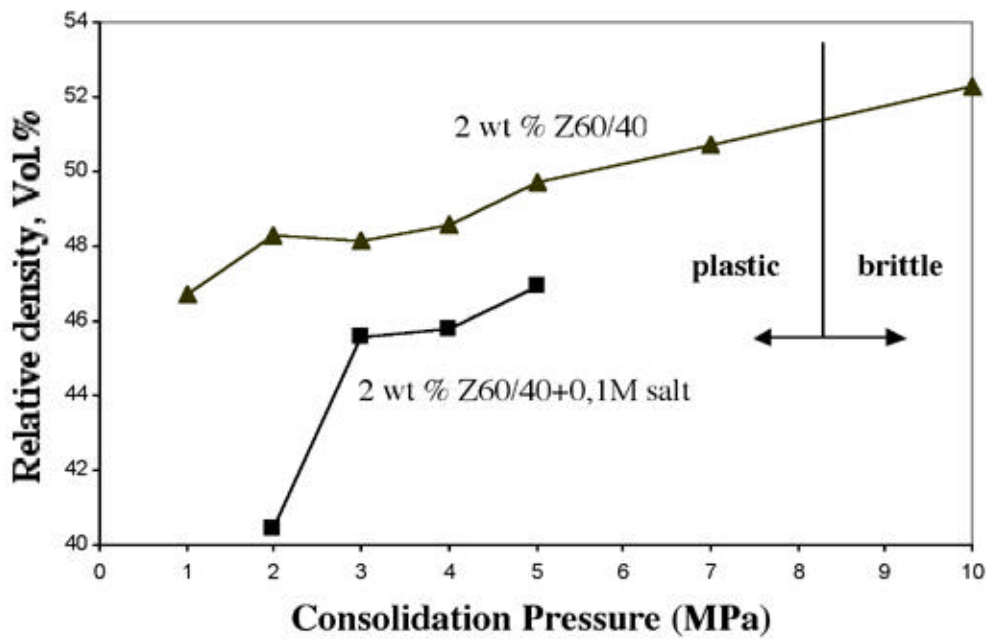


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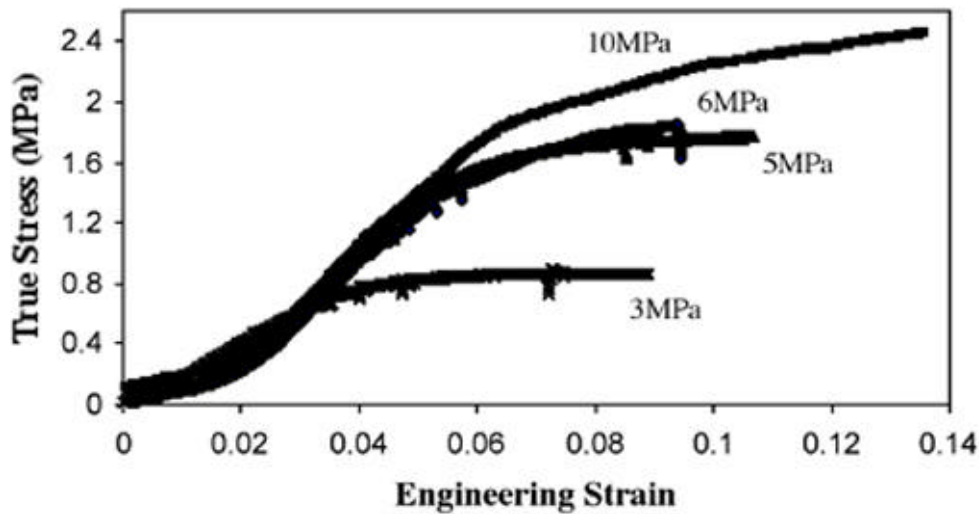


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